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Indian Standard

SPECIFICATION FOR β-CHLOROETHYLAMINE HYDROCHLORIDE SOLUTION

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Indian Standard
SPECIFICATION FOR β-CHLOROETHYLAMINE HYDROCHLORIDE SOLUTION

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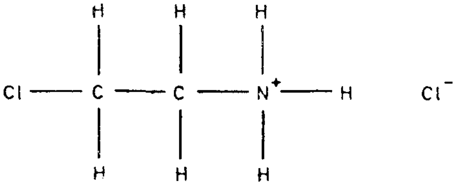
Indian Standard

SPECIFICATION FOR β-CHLOROETHYLAMINE HYDROCHLORIDE SOLUTION

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 July 1979, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 β-Chloroethylamine hydrochloride is an important dye intermediate for the manufacture of reactive dyes. It is normally derived by chlorination of 2-ethanolamine. β-Chloroethylamine is isolated as its hydrochloride and is sold in the form of 60 percent (m/m) aqueous solution. It is represented by the following structural formula:



$$\mathrm{Cl{-}CH_2{-}CH_2{-}NH_3^+\ Cl^-}$$

β-CHLOROETHYLAMINE HYDROCHLORIDE

(Molecular Mass 115·9)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for β-chloroethylamine hydrochloride solution.

---

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a clear pale yellow solution and free from sediments.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR β-CHLOROETHYLAMINE HYDROCHLORIDE SOLUTION

(*Clauses* 2.2, 4.3.1, 4.3.2 *and* 5.1)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix)
(1)	(2)	(3)	(4)
i)	Assay*		
	a) Based on hydrolyzable chlorine content, percent by mass, *Min* (X)	60	A-1
	b) Based on nitrogen estimation, percent by mass, *Max* (Y) *Min*	$X + 3$ 60	A-2
ii)	Relative density at 27°C	1·210 to 1·225	A-3

*The assay based on nitrogen estimation always gives a higher value (Y) than that based on the hydrolyzable chlorine content (X) as it does not take into account the extent of hydrolysis of the product nor, the presence of unreacted 2-ethanolamine. It may be used as countercheck to assess the presence of 2-ethanolamine, too much of which is objectionable. The difference between these two figures (Y and X) shall not be more than 3 percent.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in glass, HDPE or PVC carboys. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number/date of manufacture;

d) Gross, tare and net mass; and

e) The minimum cautionary notice worded as under:

‘ POISONOUS, AVOID CONTACT WITH SKIN ’.

3.2.1 The container may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics, namely relative density shall be conducted on composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (*see* **4.2.2**), the result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Test shall be carried out according to the methods prescribed in Appendix A as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

Note — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHOD OF TEST FOR β-CHLOROETHYLAMINE HYDROCHLORIDE SOLUTION

A-1. ASSAY, BASED ON HYDROLYZABLE CHLORINE CONTENT

A-1.0 Outline of the Method — The hydrolyzable chlorine is converted into ionizable chlorine by substitution with morpholine and the total ionizable chlorine is determined potentiometrically by titration with standard silver nitrate solution. The ionizable chlorine is also determined separately without hydrolyzing and by deducting this figure from the total chlorine content, the estimation of hydrolyzable chlorine content may be made.

A-1.1 Reagents

A-1.1.1 *Standard Silver Nitrate Solution* — 0·1 N.

A-1.1.2 *Dilute Sulphuric Acid*

A-1.1.3 *Morpholine*

A-1.1.4 *Dioxane*

A-1.2 Procedure

A-1.2.1 *Total Hydrolyzable Chlorine Content* (*T*)

A-1.2.1.1 Weigh, accurately 0·3 to 0·4 g of the material into 250-ml reflux flask. Add 50 ml dioxane and 10 ml morpholine. Allow the mixture to stand for 30 minutes; shake the mixture at the intervals of five minutes. Attach a water condenser and reflux the mass for two hours. Cool the mass to room temperature and transfer it to a 500-ml beaker. Rinse the flask thrice with distilled water (25 ml for each washing), add 5 ml of dilute sulphuric acid and titrate potentiometrically with silver nitrate solution.

A-1.2.1.2 *Calculation*

(1 ml of 0·1 N $AgNO_3$ = 0·003 55 g of Cl)

Total chlorine content, per g

$$(T) = \frac{V \times 0{\cdot}003\,55 \times N}{0{\cdot}1 \times M_1}$$

where

V = volume in ml of silver nitrate solution used for titration,

N = normality of silver nitrate solution, and

M_1 = mass in g of sample taken for estimation.

A-1.2.2 *Ionizable Chlorine Content (I)*

A-1.2.2.1 Weigh accurately about 0·5 g of sample and transfer to a 250-ml beaker. Add 50 ml of distilled water and 5 ml of dilute sulphuric acid and titrate potentiometrically with silver nitrate solution.

A-1.2.2.2 *Calculation*

$$\text{Ionizable chlorine content (I), per g} = \frac{V \times 0{\cdot}00355 \times N}{0{\cdot}1 \times M_2}$$

where

V = volume in ml of silver nitrate solution used for titration,

N = normality of silver nitrate solution, and

M_2 = mass in g of sample taken for estimation.

A-1.2.3 *Hydrolyzable Chlorine Content (H)*

A-1.2.3.1 *Calculation* — Calculate the hydrolyzable chlorine content as follows:

$$\text{Hydrolyzable chlorine content } (H) = (T - I) \text{ per g}$$

where

T = total hydrolyzable chlorine content per g as determined in **A-1.2.1.2**, and

I = ionizable chlorine content per g as determined in **A-1.2.2.2**.

A-1.2.4 *Assay, Based on Hydrolyzable Content*

A-1.2.4.1 *Calculation* — Calculate the assay as follows:

$$\text{Assay (based on hydrolyzable chlorine content), percent by mass } (X) = \frac{(H) \times 115{\cdot}93 \times 100}{35{\cdot}5}$$

where

H = hydrolyzable chlorine content per g as determined in **A-1.2.3.1**.

A-2. ASSAY BY NITROGEN ESTIMATION

A-2.0 Outline of the Method — The sample is digested in sulphuric acid so that the amino-nitrogen can be isolated as ammonia and estimated volumetrically.

A-2.1 Reagents

A-2.1.1 *Standard Hydrochloric Acid Solution* — 0·1 N.

A-2.1.2 *Sodium Sulphate* — anhydrous.

A-2.1.3 *Red Mercuric Oxide*

A-2.1.4 *Concentrated Sulphuric Acid*

A-2.1.5 *Sodium Hydroxide Solution* — 40 percent (*v/v*).

A-2.1.6 *Sodium Thiosulphate Solution* — 10 percent.

A-2.1.7 *Boric Acid Solution* — 5 percent.

A-2.2 Procedure

A-2.2.1 Weigh, accurately, about 0·2 to 0·3 g of sample in 500-ml Kjeldahl's flask, add sodium sulphate, 0·3 g mercuric oxide and 25 ml of concentrated sulphuric acid. Digest the mixture till it becomes clear and colourless. Again digest the mixture for further four hours. Cool the flask to room temperature, add 200 ml of water and cool to 5°C in ice-water-bath. Add 200 ml of 40 percent sodium hydroxide solution at 5°C, followed by 10 ml of 10 percent sodium thiosulphate solution. Set the flask for distillation. Collect about 200 ml distillate in a 500-ml conical flask containing 10 ml of 5 percent boric acid solution.

Titrate the distillate with hydrochloric acid solution using methyl red as an indicator.

A-2.2.2 *Blank Determination* — Repeat the same experiment without taking sample under test.

A-2.3 Calculation

Assay (based on nitrogen estimation), percent by mass

$$(Y) = \frac{(V_1 - V_2) \times N \times 115{\cdot}93 \times 100}{M_3 \times 1\,000}$$

where

V_1 = volume in ml of hydrochloric acid used when the sample was digested in sulphuric acid,

V_2 = volume in ml of hydrochloric acid used for blank determination,

N = normality of hydrochloric acid, and

M_3 = mass in g of sample taken for estimation.

A-3. RELATIVE DENSITY

A-3.1 Determine the relative density of the material at 27°C as prescribed in **4** of IS : 5299-1969*.

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*Methods of sampling and tests for dye intermediates.